WORKING DRAFT FOR DISCUSSION
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Draft - What do we need a procedure to do?

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The details of the following text were developed without review by other FACDQ TWG, PWG, or FACDQ members and do not necessarily represent a consensus of these groups. The text was prepared based on discussions of the PWG and at the request of Triangle Associates. This text was also prepared within the context of developing a pilot study to test procedures, not necessarily to define the final procedures for the FACDQ.

Introduction

The Policy Work Group was asked to respond to the question, "What do we want a procedure to do?" The starting point for responding to this question was a concept paper that Jim Pletl prepared and the Policy Work Group discussed during its January 23, 2006 call.

Following the January 23 call, there was considerable discussion via email among Policy Work Group members regarding this topic. A subgroup was convened to prepare a document for the February 6 Policy Work Group call. The subgroup reviewed the emails and prepared a draft for the February 6 Policy Work Group call.

The Policy Work Group met by conference call on February 27 and discussed a new draft prioritized list of "needs" for a procedure. This draft was prepared by Dick Reding, Mary Smith and Jim Pletl. The intent of this draft was to focus MQO discussions only on the issues of greatest importance to the FACDQ because the number of potential issues requiring MQOs for the pilot study would likely preclude pilot testing this year. Comments were received from the Policy Work Group and the prioritized list of procedure issues was amended accordingly. The amended list was presented to the Technical Work Group on March 1 for their comments and discussion.

Since the prioritized list of objectives differs from the objectives that were listed in the draft "What do we want a procedure to do?" document, it was agreed that a document should be developed to support the new prioritized list of objectives. This document attempts to characterize each prioritized objective and provide suggestions of how each

objective could be evaluated for each procedure. Each objective is followed by a number in parentheses; this is the number of the characteristic in the procedure matrix that most closely represents the objective in question. The term "limit" is used generally to refer to detection and quantitation limits since the FACDQ has not yet defined them. Examples of how to measure specific objectives are also sometimes written broadly and may not apply in every case (Lc, Ld, Lq, other).

The procedure(s) must:

1. **provide an explicit estimate of bias for limits and this estimate must match that observed in labs at those limits. (1)**

   evaluated by:

   a. reviewing procedure(s) and specifically identifying the quantitative measure of bias predicted for each limit.
   b. requiring labs to analyze blind samples (blanks or spikes) and comparing observed bias to that cited by the procedure(s), respectively.

2. **provide an explicit estimate of precision for limits and this estimate must match that observed in labs at those limits. (2)**

   evaluated by:

   a. reviewing procedure(s) and specifically identifying the quantitative measure of precision predicted for each limit.
   b. requiring each lab to analyze multiple blind samples (blanks or spikes) and comparing precision among those samples to that cited by the procedure(s), respectively.

3. **provide an explicit false positive rate for limits and this rate must match that observed in labs at those limits. (3)**

   evaluated by:

   a. reviewing procedure(s) and specifically identifying the false positive error rate predicted for each limit.
   b. comparing the false positive rate of lab blanks at the levels of Lc and Ld to those predicted by the procedure(s).

c. comparing the false positive rate of concluding a concentration is $\geq$ Lq when the concentration is < Lq (by analyzing samples spiked at levels less than Lq) to that predicted by the procedure(s).

4. provide an explicit false negative rate for limits and this rate must match that observed in labs at those limits. (4)

evaluated by:

a. reviewing procedure(s) and specifically identifying the false negative error rate predicted for estimates like Lc, Ld and/or Lq.
b. comparing the false negative rate of results obtained by analyzing samples spiked at the limit concentration to those predicted by the procedure(s), respectively.

5. require that qualitative identification takes place at the determined detection or quantitation limits. (5)

evaluated by:

a. (*Need help here from those much more familiar with this issue)*
b. reviewing the procedure(s) and determining whether they define the analytical technologies where qualitative identification takes place.
c. reviewing the procedure(s) and determining if they define specific measures which are used to document qualitative identification.
d. reviewing the procedure(s) to determine if qualitative identification is required at each limit (Lc, Ld, Lq, other).

6. adequately represent variability in lab performance. (8, 9, 10)

evaluated by determining whether the procedures:

a. use data to calculate limits that are collected over enough time to capture variability in performance relative to MQOs
b. recalculate limits at a frequency that captures variability in performance relative to MQOs
c. incorporate variability due to the use of multiple instruments/lab
d. incorporate variability due to use of multiple analysts/lab
e. incorporate variability occurring across laboratories.
f. address varying analyte recovery

g. provide recommendations or limit choices for outlier tests
h. address varying numbers of different concentrations (spikes) that can be used between laboratories
i. address varying numbers of replicates per concentration (spike) that can be used between laboratories
j. address varying combinations of concentrations (spikes) that can be used between laboratories.
k. adequately accommodate different models of instruments used per analyte and technology to calculate limits

7. be capable of calculating limits using matrices other than lab reagent grade water. (11)

evaluated by:

a. reviewing procedures and determining that there is nothing precluding the use of matrices other than reagent grade water to calculate limits.
b. reviewing procedures to determine if they incorporate steps to illustrate when limits adopted for an analytical method can not be met in a matrix other than lab reagent grade water.
c. reviewing procedures to determine if they provide instructions on preparing an analyte-free matrix that approximates the matrix in question.

8. only use data that results from test methods conducted in their entirety. (12)

evaluated by determining whether the procedure(s):

a. require that samples used to calculate detection and quantitation limits undergo all steps outlined in an analytical method (prep method, extraction, etc.)
b.

9. explicitly adjust or account for situations where method blanks always return a non-zero result/response. (14)

evaluated by:

a. reviewing the procedure(s) and determining if they identify which analytical methods/analyte combinations always return a non-zero result/response

b. reviewing the procedure(s) and determining if they require calculation of statistics regarding non-zero results/responses.
c. reviewing the procedure(s) and determining if they mathematically adjust limits for non-zero results/responses.

10. explicitly adjust or account for situations where method blanks are intermittently contaminated. (15)

evaluated by:

a. reviewing the procedure(s) and determining if they characterize and define analytical method/analyte combinations where intermittent contamination can be expected to occur.
b. reviewing the procedure(s) and determining if they define intermittent contamination.
c. reviewing the procedure(s) and determining if they mathematically adjust limits when method blanks are intermittently contaminated.

11. be clearly written with enough detail so that most users can understand and implement them. (Procedural Complexity, 23-27)

evaluated by:

a. asking users to interpret data prior to our after-procedure calculations are carried out. Examples could include "What is the resulting detection limit?", "What is the resulting quantitation limit?", "What is the blank bias?
b. asking users questions about the procedure characteristics, using the matrix as a point of reference. Examples could include "Do the procedures address recovery?", "How often is a limit calculated by the user?", or "How often is data generated to calculate limits for a given procedure?".
c. testing users to perform calculations or run software and interpret results.
d. asking users to select spikes for given circumstances.
e. reviewing the procedure(s) and determining which ones minimize the amount of data required to calculate analytical limits beyond that normally generated by analytical methods.
f. determining that the procedure(s) do not require skills of users in addition to those that are normally required by laboratories.

12. be cost effective. (28)

evaluated by:

a. reviewing the procedure(s) and determining which ones minimize the amount of data required to calculate analytical limits beyond that normally generated by analytical methods.
b. determining whether the procedure(s) require the purchase of software or equipment in addition to that which is normally required by laboratories.
c. determining that the procedure(s) do not require skills of users in addition to those that are normally required by laboratories.

13. be applicable to all users and test methods. (36)

evaluated by:

a. testing procedures against other objectives among a representative sample of users (labs, states, EPA, accreditation programs, etc.)
b. testing procedures against other objectives among a representative sample of analytical test methods (different technologies and analytes, for example)